

What is claimed is:

- 5 1. A process for removing trioxane from a mixture I of formaldehyde, trioxane and water, by
  - 10 a) distilling the mixture I in a first distillation stage at a pressure of from 0.1 to 2 bar to obtain a stream II which comprises formaldehyde and a stream III which comprises predominantly trioxane and additionally water and formaldehyde,
  - 15 b) mixing the stream III with a recycle stream VII which comprises predominantly trioxane and additionally water and formaldehyde to obtain a stream IIIa which comprises predominantly trioxane and additionally water and formaldehyde,
  - 20 c) distilling the stream IIIa, if appropriate after removing low boilers from the stream III or IIIa in a further distillation stage, in a second distillation stage at a pressure of from 0.2 to 10 bar, the pressure in the second distillation stage being at least 0.1 bar higher than the pressure in the first distillation stage, to obtain a stream IV of trioxane and a stream V which comprises predominantly trioxane and additionally water and formaldehyde,
  - 25 d) distilling the stream V in a third distillation stage at a pressure of from 0.1 to 4 bar to obtain a stream VI which comprises predominantly water and additionally formaldehyde, and the recycle stream VII which comprises predominantly trioxane and additionally water and formaldehyde,
  - 30 e) if appropriate, distilling the stream VI in a fourth distillation stage to obtain a stream VIII which comprises predominantly water, and a stream IX which comprises predominantly formaldehyde.
- 35 2. The process according to claim 1, wherein the pressure in the second distillation stage is from 0.5 to 10 bar higher than the pressure in the first distillation stage.
- 40 3. The process according to claim 1 or 2, wherein the first distillation stage is carried out at a pressure of from 0.5 to 2 bar and the second distillation stage at a pressure of from 2.5 to 8 bar.

4. The process according to any of claims 1 to 3, wherein the third distillation stage is carried out at a pressure of from 0.1 to 1 bar.
5. The process according to any of claims 1 to 4, wherein the first distillation stage is carried out in a distillation column having at least two theoretical plates, the second distillation stage in a distillation column having at least 2 theoretical plates and the third distillation stage in a distillation column having at least one theoretical plate.
6. The process according to any of claims 1 to 5, characterized by the following composition of streams I–VII:
- stream I: from 35 to 80% by weight of formaldehyde, from 25 to 45% by weight of water, from 1 to 30% by weight of trioxane;
- stream II: from 51 to 80% by weight of formaldehyde, 20 to 49% by weight of water, 0 to 1% by weight of trioxane;
- stream III: from 1 to 15% by weight of formaldehyde, 15 to 35% by weight of water, 60 to 80% by weight of trioxane;
- stream IIIa: from 3 to 20% by weight of formaldehyde, 10 to 30% by weight of water, 60 to 80% by weight of trioxane;
- stream IV: from 95 to 100% by weight of trioxane, 0 to 5% by weight of water and secondary components;
- stream V: from 5 to 20% by weight of formaldehyde, 15 to 35% by weight of water, 50 to 80% by weight of trioxane;
- stream VI: from 10 to 25% by weight of formaldehyde, 75 to 90% by weight of water, 0 to 1% by weight of trioxane;
- stream VII: from 5 to 20% by weight of formaldehyde, 10 to 30% by weight of water, 60 to 80% by weight of trioxane,
- and the streams I, III, IIIa, V and VII may also contain up to 15% by weight of low boilers selected from the group consisting of methyl formate, methylal, dimethoxydimethyl ether, trimethoxydimethyl ether, methanol, formic acid, and also further hemiacetals and full acetals.

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7. The process according to any of claims 1 to 6, wherein a low boiler removal is undertaken in a further distillation stage between the first and the second distillation stage.
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8. A process for preparing trioxane from an aqueous formaldehyde solution, by feeding a stream X of an aqueous formaldehyde of a trioxane synthesis stage and converting it under acidic conditions to obtain the mixture I, and removing trioxane from the steam I by the process according to any of claims 1 to 6.
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9. The process according to claim 8, wherein a separate trioxane synthesis stage is carried out and precedes the first distillation stage.
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10. The process according to claim 8, wherein the trioxane synthesis stage and the first distillation stage are carried out together as a reactive distillation.
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11. The process according to any of claims 8 to 10, wherein the stream X contains from 55 to 85% by weight of formaldehyde and from 15 to 45% by weight of water.
12. The process according to any of claims 8 to 11, wherein the stream X is obtained from an aqueous formaldehyde solution of low formaldehyde concentration by concentrating in an evaporator.
13. The use of trioxane, preparable by the process according to any of claims 8 to 12, for preparing polyoxymethylene (POM), polyoxymethylene derivatives and diaminodiphenylmethane (MDA).